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<p>(21) International Application Number: PCT/EP94/01848 (22) International Filing Date: 4 June 1994 (04.06.94) (30) Priority Data: 9311536.8 4 June 1993 (04.06.93) GB (71) Applicant (for AU BB CA GB IE LK MN MW NZ SD TT only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA GB IE LK MN MW NZ SD TT): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (72) Inventors: BEE, Rodney, David; 10 Stevenson Court, Eaton Ford, St Neots, Cambridge PE19 3LF (GB). HARRIS, Clive, Stanbra; "Vaun", Chapple Lane, Souldrop, Bedford MK44 1HB (GB). WARBOYS, Michael, John; 8 Roland Way, Higham Ferrers, Wellingborough, Northampton NN9 8EQ (GB). (74) Agent: ROSCOE, Brian, Corrie; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).</p>		<p>(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, GE, HU, JP, KG, KP, KR, KZ, LK, LU, LV, MD, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TJ, TT, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: HOT SOLUBLE EXTRACTABLE FOOD PRODUCT AND PROCESS FOR PREPARING SAME (57) Abstract The invention relates to a hot soluble extractable food product such as an instant tea or coffee comprising freeze dried granules that are prepared from an extract containing no more than about 25 % by weight food solids, have a bulk density of no more than about 80 g/l and a Friability Index of less than about 40 % (preferably less than about 30 %, especially less than about 25 %). The term Friability Index is defined and a method of determining same is described. A process for preparing a hot soluble extractable food product is also described which comprises: extracting an extractable food, cooling the extract, aerating the extract with a gas comprising a gas or mixture of gases substantially more soluble than nitrogen or a mixture of nitrogen and a gas or mixture of gases substantially more soluble than nitrogen to produce a foam, substantially freezing than foam, shattering it into granules, and freeze drying the granules to yield the product.</p>		

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HOT SOLUBLE EXTRACTABLE FOOD PRODUCT
AND PROCESS FOR PREPARING SAME

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Field of the Invention

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This invention relates to a low density but attrition-resistant hot soluble extractable food product, specifically freeze dried tea or coffee granules, and a process for preparing same.

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Background of the Invention

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With the exception of water, tea is the most widely consumed of all beverages. In fact, the worldwide per capita consumption has been estimated at 0.1 litre per day. Coffee is another beverage that is consumed in great volumes around the world. But while the invention is suitable for manufacturing low density and attrition-resistant coffee granules, the following description generally refers to tea.

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Most of the tea consumed daily is so called black tea which is obtained by harvesting new leaves of the plant *Camellia sinensis* and withering, rolling, enzymatically oxidizing, firing and sorting them.

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In Western countries consumers tea is generally sold in bags which are placed in hot water and allowed to stew but discarded prior to drinking the tea. However, more recently tea can also be prepared by dissolving a powdered

or granulated tea product in hot water thus avoiding the need to handle and dispose of soggy bags. This powdered or granulated tea is known as instant tea and consists of the hot soluble product of black tea leaves that have been
5 extracted, concentrated and dried. Flavours may be added or components such as caffeine may be removed during this process to meet the tastes and demands of consumers.

The freeze dried tea granules typically prepared by known
10 methods tend to be prepared from an extract of high solids concentration, for example 20-60%, which provides a density of about 90-100 g/l. This is equivalent to a weight per teaspoon of about 0.5 g of tea solids. Consumers in the United Kingdom typically enjoy their tea at a strength of
15 about 0.5 grams of tea solids per cup and may add milk or cream so it is convenient to simply add one teaspoon of granules to a cup of hot water. However, consumers on the European continent generally prefer their tea to be weaker, typically about 0.35 grams of tea solids per cup, and tend
20 not to add any milk. Consequently, continental tea drinkers would find one teaspoon of tea granules weighing 0.5 g provides an unacceptably strong cup of tea. The easy answer to this problem would be for them to use less than one teaspoonful but consumers would find it difficult to
25 measure out their preferred volume of granules each time in order to prepare a cup of tea that suits their taste and also inconvenient so that answer is not an appropriate solution.

30 Ideally, consumers would prefer to simply add one teaspoon of tea product to one cup of hot water and so if the volume of tea is to remain constant the amount of tea per teaspoon or the density of the granules in terms of the tea content would need to decrease from about 0.5 grams per teaspoon,
35 that is 90-100 g/l, to about 0.35 grams per teaspoon, that is about 60 g/l.

Unfortunately, while it is possible to produce tea granules with a density of about 60 g/l by known methods these granules show poor resistance to attrition and therefore readily break down into dust. Consumers would perceive such a product as being of poor quality so that is not an acceptable solution to the problem either.

USP 3,749,378 (General Foods) discloses an apparatus for foaming coffee or tea extracts and emulsions using carbon dioxide, nitrous oxide or more preferably nitrogen to a certain density which can then be freeze or spray dried. However, according to the specification, carbon dioxide does not give the same low bulk density products at the same initial foam density of nitrogen. The products that might be obtained by the apparatus are not characterised. Apparently spray dried coffee granules having a bulk density of 52 g/l can be prepared when foaming with nitrogen but there is no indication as to their friability. Furthermore, freeze drying is discouraged on a cost basis.

However, the present inventors have developed a novel process for preparing freeze dried extractable food granules from extracts having a low solids concentration, for example 10-25%, giving a product density of 50-80 g/l and which are characterised by an attrition-resistant structure. Furthermore, insofar as tea is concerned the granules are darker in colour and look more tea-like than tea granules prepared by known methods. The inventors obtained similar results using coffee rather than tea.

Definition of the Invention

The invention may be said in broad terms to relate to a hot soluble extractable food product comprising granules characterised in that the granules are prepared from an

extract containing less than about 25% soluble food solids, have a bulk density of no more than about 80 g/l and a Friability Index (as herein described) of less than about 40%.

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The Friability Index of a granule refers to the tendency of that granule to break down by attrition. This term is defined below along with a method of accurately and reproducibly quantifying same.

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Preferably the extractable food is tea or coffee and the granules are freeze dried.

15

Preferably the liquor contains between about 10 to about 25% or especially about 16 to about 21% soluble food solids prior to freeze drying.

20

Preferably the granules have a bulk density of about 50 to about 80 g/l or especially about 60 to about 70 g/l and a Friability Index less than about 30% or especially less than about 25%.

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The invention in broad terms may also be said to relate to a process for preparing a hot soluble extractable food product comprising:

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- (a) extracting an extractable food product,
- (b) cooling the extract and aerating it with a gas comprising:

- (i) a gas or mixture of gases substantially more soluble than nitrogen; or
- (ii) a mixture of nitrogen and a gas or mixture of gases substantially more soluble than nitrogen,

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- to produce a partially frozen foam,
- (c) substantially freezing the foam,
- (d) shattering the substantially frozen foam into

granules, and

(e) freeze drying the granules to yield the hot soluble product.

5 Preferably the extractable food is tea or coffee and is concentrated to between about 10 and about 25% or especially between about 16% to about 21% tea or coffee solids by weight and kept at a temperature of about 10°C prior to the cooling and aerating. The cooling and
10 aerating is preferably carried out substantially simultaneously in one unit such as an ice cream freezer or other art-known apparatus. Preferably the cooling involves lowering the extract to a temperature of no less than about -5°C.

15 The extract is preferably aerated with carbon dioxide, nitrous oxide, a mixture of those, or a mixture of nitrogen and either or both carbon dioxide and nitrous oxide. It is also preferred that the foam is cooled in liquid nitrogen and the frozen mass thus obtained shattered into granules
20 smaller than about 5 mm in diameter by a hammer mill or the like.

25 Description of the Invention

The invention will now be described in detail with reference to the following photographic representations, but it should be understood that they are not intended to
30 define the scope of the invention in any way:

Figure 1 is a photographic representation of freeze dried low density tea granules that have been prepared by foaming a concentrated tea extract with nitrogen.

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Figure 2 is a photographic representation of freeze dried low density tea granules that have been prepared by foaming

a concentrated tea extract with carbon dioxide.

Figure 3 is a photographic representation of a scanning electron micrograph of a freeze dried low density tea granule that has been prepared by foaming a concentrated tea extract with nitrogen.

Figure 4 is a photographic representation of a scanning electron micrograph of a freeze dried low density tea granule that has been prepared by foaming a concentrated tea extract with carbon dioxide.

Tea liquor is extracted from tea leaves by art known means to give a tea solids concentration of typically 3 to 12%. This is concentrated by art known means to less than about 25% by weight tea solids, preferably between about 10 and about 25%, or more preferably between about 16% to 21% by weight tea solids.

The concentrated tea extract is preferably brought to a temperature of about 10°C then cooled, for example to a temperature not less than about -5°C, and aerated, perhaps substantially simultaneously in one art-known apparatus such as an ice cream freezer, with a gas comprising: (i) a gas or mixture of gases substantially more soluble than nitrogen; or (ii) a mixture of nitrogen and a gas or mixture of gases substantially more soluble than nitrogen. Gases substantially more soluble than nitrogen (2.35 cm³ in 100 cm³ H₂O at 0°C) include carbon dioxide and nitrous oxide (179.7 cm³ and 130.52 cm³ in 100 cm³ H₂O at 0°C respectively). Foaming with carbon dioxide alone is especially preferred.

The foamed concentrated extract is substantially frozen, preferably rapidly, by art known means such as by immersion into liquid nitrogen and the frozen mass thus produced is

shattered in to granules smaller than about 5 mm in diameter on average using a hammer mill or the like. These granules are then freeze dried to yield a low density product, that is typically having a bulk density of about 50 to about 80 g/l but preferably of about 60 to about 70 g/l.

The granules are also characterised by being less friable or more ready to resist attrition than hot soluble extractable vegetable food granules manufactured by the methods described in the prior art.

The friability of a granule depends on the nature of the applied forces as well as the intrinsic mechanical properties of the granule itself. That is why instant tea granules despite being less dense than similar granules such as those of coffee do not necessarily have a lower compressive strength.

Several methods of measuring friability are known but there is no absolute measure of friability. This is because different friability tests break down granules in different ways and therefore apply different types and degrees of force. The intrinsic mechanical properties of the material and the structure of the granule are also very important.

The size and shape of granules usually differ widely which complicates things further. Friability can be measured by testing a given mass or volume of granules. But it is often difficult to relate the results from such tests to single granule properties as packing and intergranule forces can greatly influence the measurements. It might therefore be desirable to perform mechanical tests on standard shaped blocks or bars so that friability measurements can be directly related to material and structural properties.

However, for the purposes of describing and defining the present invention the friability of granular material, specifically extractable food products such as tea and coffee, is based on the proportion of fines, that is granules having a diameter of less than 1 mm, that are generated when a known mass of freshly granulated material is subjected to the sort of abrasive forces they might experience during further processing, packing and transportation. This parameter is expressed as a percentage and termed the "Friability Index".

A low Friability Index indicates that a granulated product is attrition resistant and is desirable to consumers as they associate fragile granules and an abundance of dust with poor quality.

The Friability Index of a granulated material as defined above is determined as follows:

- (1) The particle size range of a given granulated product is ascertained by vibrating a 50 g sample of same on a sieve stack at an amplitude of 0.58 mm for 2 minutes. The sieve stack comprises four sieves that comprise mesh sizes of 5 mm, 2.5 mm, 2.0 mm and 1.0 mm respectively;
- (2) the granules retained on the 2.5 mm mesh are re-sieved for 5 minutes at an amplitude of 1.55 mm;
- (3) the granules that have passed through the 1.00 mm mesh, that is the fines, and the granules that have been retained on the 2.5 mm sieve after the re-sieving are weighed separately; and
- (4) the Friability Index is calculated as the percentage of the mass of the fines with respect to the mass of the granules retained on the 2.5 mm sieve after the re-sieving, ie:

the mass of granules having
Friability Index = $\frac{\text{a diameter less than 1.0 mm}}{100} \times$

5 the mass of granules having
a diameter between 2.5 and 5 mm

The Friability Index of granules according to the present invention is less than 40%, preferably less than 30% and
10 more preferably less about 25%.

The unique structural characteristics of the attrition-resistant granules of the present invention affects their appearance to the naked or partially aided eye. This is
15 evident by comparing the freeze dried granules shown in Figure 1 with those shown in Figure 2.

The freeze dried granules shown in Figure 1 were prepared by aerating a concentrated tea extract with nitrogen
20 whereas the freeze dried granules shown in Figure 2 were prepared by aerating a similarly concentrated tea extract with carbon dioxide. The granules shown in Figure 2 have a "crisper", more glassy appearance than those shown in Figure 2. The pore sizes are greater. The granules are
25 also slightly darker in colour (this is more noticeable when viewed by the naked eye). This darker colour is considered desirable by consumers as it suggests a richer and higher quality product.

30 The differences in the microscopic structures of granules prepared by aerating carbon dioxide rather than nitrogen are apparent when comparing the scanning electron micrographs of each shown in Figures 4 and 3 respectively. The microscopic structure of the granule prepared by
35 nitrogen aeration shown in Figure 3 at a magnification of 200X resembles a brittle sponge-like structure wherein the solid portions are highly pitted. This explains why small

parts of the granules readily break away to form dust. In contrast to this, while the microscopic structure of the granule prepared by carbon dioxide aeration shown in Figure 4, also at a magnification of 200X, also resembles a sponge-like structure, the solid portions much smoother and almost free of pitting. This granule structure is more resistant to attrition; less of the structure breaks off to form dust.

The invention will now be described in detail with reference to the following experimental examples, but it should be understood that they are not intended to define the scope of the invention in any way:

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20

EXAMPLES5 Example 1

10 A tea extract was obtained from black tea and concentrated to 16% tea solids by weight at a temperature of 10°C. This extract was divided into two substantially equal portions which were brought to 10°C then foamed with nitrogen gas and carbon dioxide gas respectively using a Crepaco type W-104G ice cream freezer equipped with a type 30 dasher until a foam density of 0.45 g/cm³ was obtained. The pressure within the freezing barrel of the freezer was maintained at 15 4 bar gauge throughout the freezing and the barrel walls were also kept refrigerated so that an extruded foamed product temperature of -2.5 to -3°C could be obtained.

20 In each case the extrudate was immersed in liquid nitrogen to stabilise the foam and the frozen mass obtained shattered using a hammer mill into granules of about 1 to 4 mm in diameter. These granules were then freeze dried and the resulting products subjected to a test of strength against attrition to determine their respective Friability 25 Indices. The test involved vibrating 2.5-4 mm granules for 5 minutes with an amplitude of 1.58 mm vertically and the Friability Index was calculated as the percentage of fines (ie average diameter < 1 mm) generated from the original mass tested.

30 The Friability Index of the nitrogen foamed product was found to be 45-49%. However, the Friability Index of the carbon dioxide foamed product was measured at 25-27% which represents a significant increase in strength against 35 attrition. Furthermore, the carbon dioxide foamed product had an "agglomerated" appearance not usually associated with freeze dried beverage products and the colour was

darker and more "tea-like". The bulk density of both products was 0.060 g/cm³.

5 Example 2

Colour measurements of dry tea granules prepared by aerating with carbon dioxide and dry tea granules prepared by aerating with nitrogen were made using a MINOLTA brand
10 colour meter and the results are shown in Table 1 below.

Table 1

15	<u>Colour Co-ordinate</u>	<u>Carbon dioxide aeration</u>	<u>Nitrogen</u>
	<u>aeration</u>		
	L	38 - 42	46 - 49
	a	14 - 15	15 - 16
20	b	20 - 28	30 - 35

According to this standard technique the colour of a
25 substance is described with reference to three co-ordinates (L, a and b) of a hypothetical colour sphere. The results clearly show that the granules prepared by aeration with carbon dioxide are darker and contain more orange and less grey than those
30 prepared by aeration with nitrogen.

Example 3

35 A coffee extract was prepared and concentrated to 16% coffee solids by weight. The concentrated extract was then cooled to 10°C, partially frozen and then aerated with

carbon dioxide gas to a foam density of 0.6 g/cm³, frozen to stabilise the foam, shattered into granules and freeze dried in the same way as the described in Example 1 above. The final product was found to have a bulk density of 0.06 g/cm³ and a friability index of 27%. Its appearance and structure also resembled the carbon dioxide foamed product obtained by the experiment described above as Example 1.

10 The foregoing describes the invention and preferred forms thereof. However it should be appreciated that one skilled in the art would readily appreciate that various modifications to the instant tea product and process for preparing same that have been described are possible and
15 therefore it should be understood that the detail of the materials, equipment and procedures that has been described has been presented solely for the purpose of providing a complete disclosure of the invention. The scope of the monopoly for which protection has been sought is defined
20 solely by the following claims.

CLAIMS

1. A hot soluble extractable food product comprising granules characterised in that the granules are prepared from an extract containing less than about 25% by weight soluble food solids, have a bulk density no greater than about 80 g/l and a Friability Index (as herein described) of less than about 40%
2. A food product according to claim 1, wherein the granules are prepared from an extract containing from about 10 to about 25%, or preferably about 16 to about 21%, by weight soluble food solids.
3. A food product according to claim 1 or 2, wherein the granules have a bulk density of about 50 to about 80 g/l or preferably 60 to about 70 g/l.
4. A food product according to any preceding claim, wherein the Friability Index of the granules is less than about 30%, or preferably less than about 25%.
5. A food product according to claim 1, wherein the extractable food is tea or coffee.
6. The use of a hot soluble extractable food product according to any preceding claim for the purpose of preparing a beverage.
7. A process for preparing a hot soluble extractable food product comprising:
- (a) extracting an extractable food product,
 - (b) cooling the extract and aerating it with a gas comprising:
 - (i) a gas or mixture of gases substantially more soluble than nitrogen; or
 - (ii) a mixture of nitrogen and a gas or mixture

of gases substantially more soluble than nitrogen,

to produce a partially frozen foam,

(c) substantially freezing the foam,

5 (d) shattering the substantially frozen foam into granules, and

(e) freeze drying the granules to yield the hot soluble product.

10 8. A process according to claim 7, wherein the solids concentration of the extract is adjusted prior to the cooling and aerating to between about 10 and about 25% by weight soluble solids.

15 9. A process according to claim 7 or 8, wherein the extract is aerated with carbon dioxide or nitrous oxide.

20 10. A process according to any one of claims 7 to 9, wherein the granules have a Friability Index (as herein defined) of less than about 40%.

11. A hot soluble extractable food product when prepared by the process according to any one of claims 7 to 10.

25 12. A hot soluble instant tea product substantially as herein defined with respect to Figure 2 or 4 or Examples 1 or 3.

30 13. A process for preparing a hot soluble extractable food product substantially as herein defined with respect to Figure 2 or 4 or any one of the Examples.

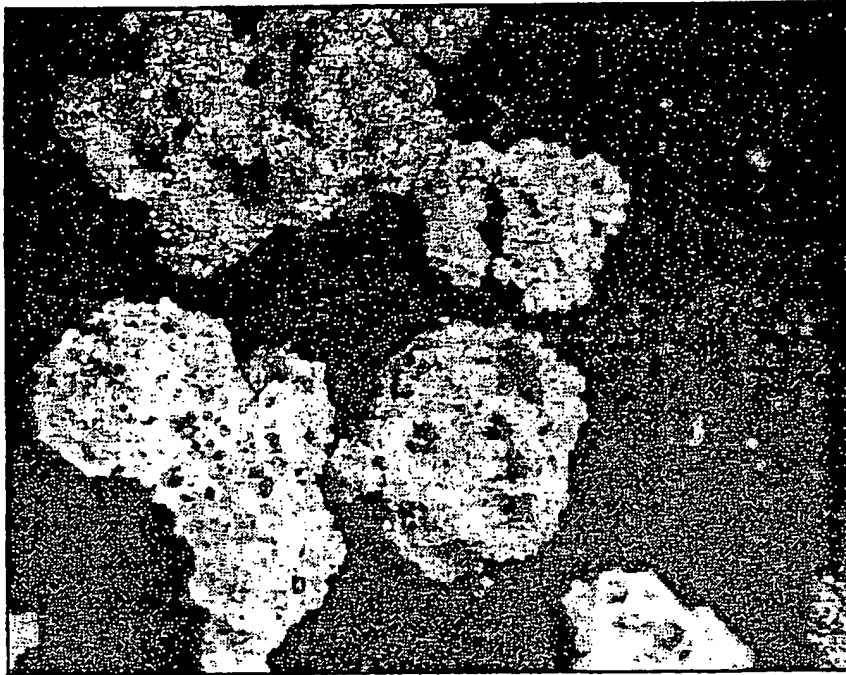


Fig. 2

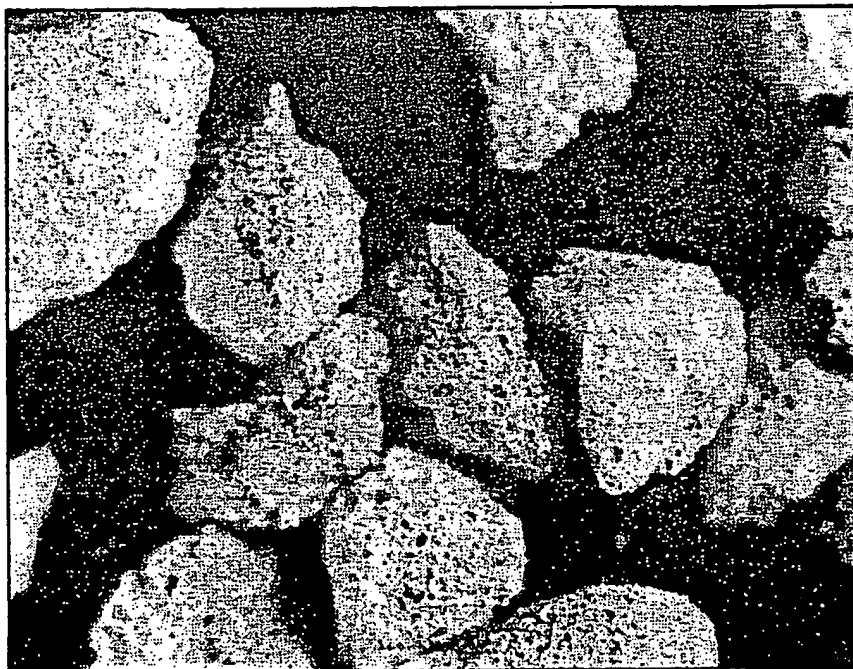
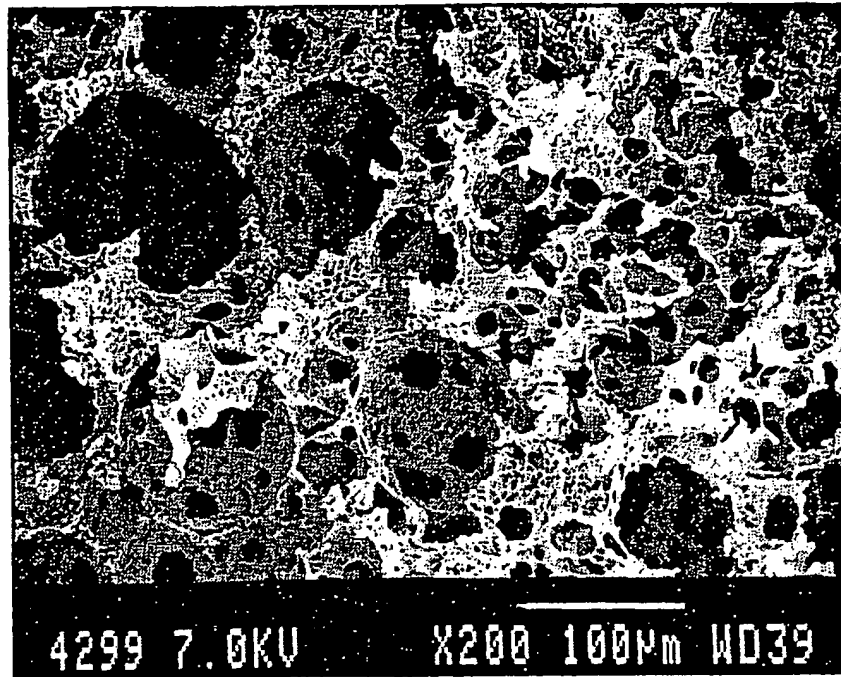
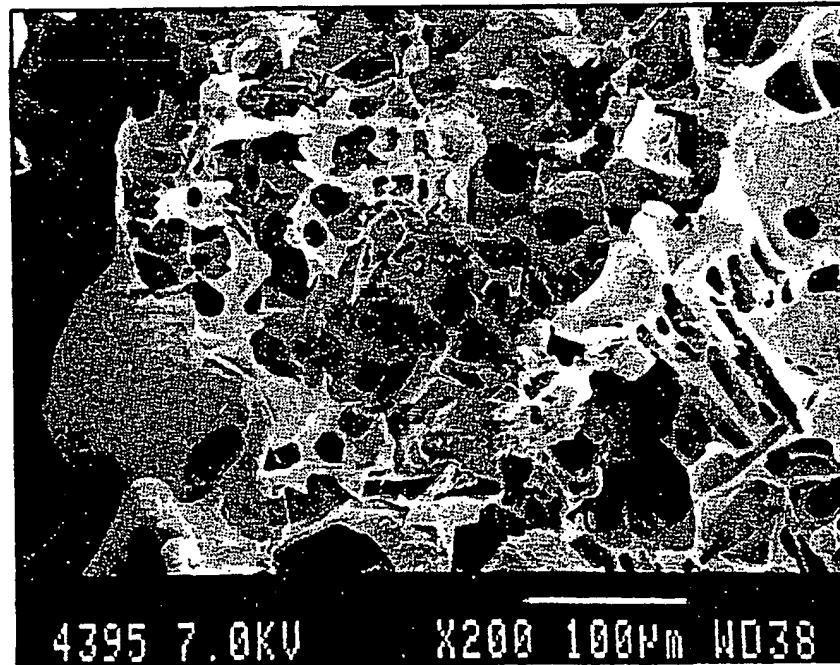


Fig. 1

*Fig. 3**Fig. 4*

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 A23F5/32 A23F5/36 A23F3/30 A23F3/26

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 A23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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